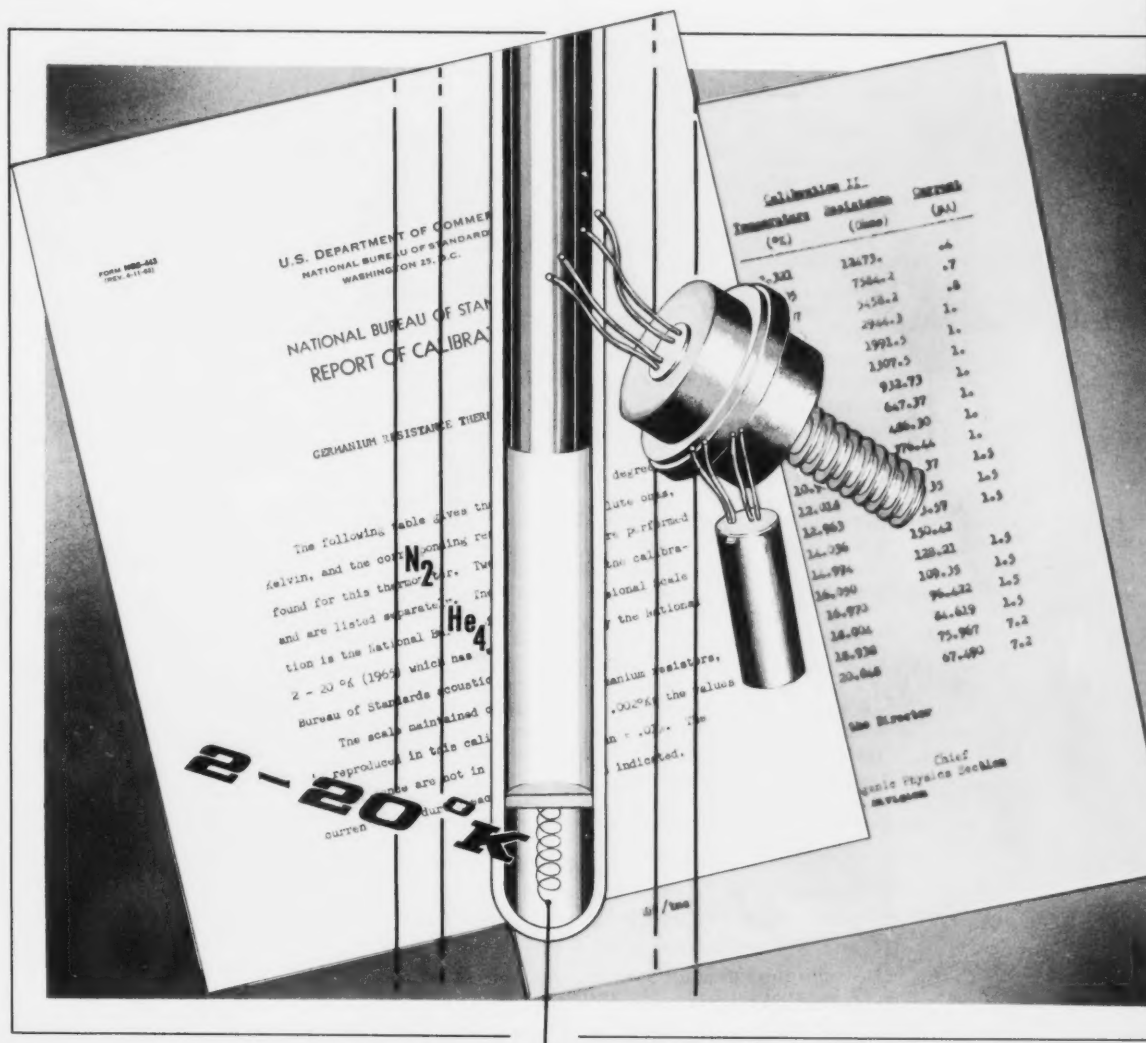


# Technical News Bulletin

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U.S. DEPARTMENT OF COMMERCE

# NATIONAL BUREAU OF STANDARDS

# Technical News Bulletin



U.S. DEPARTMENT OF COMMERCE  
John T. Connor, Secretary  
NATIONAL BUREAU OF STANDARDS  
A. V. Astin, Director

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COVER: With the recent establishment of a low-temperature scale from 4 to 14 °K, the Bureau also has initiated a calibration service for germanium resistance thermometers from 2 to 20 °K. The new scale and the calibration service are based on the acoustical thermometer in which the speed of sound in helium gas is used to determine the temperature. The new scale bridges the gap between the lower limit (10 °K) of the NBS 1955 Provisional Scale and the temperatures defined by the T<sub>88</sub> Helium<sup>4</sup> Vapor Pressure Scale (2 to 5 °K). (See story on page 102.)



## Retards Semiconductor Film Growth

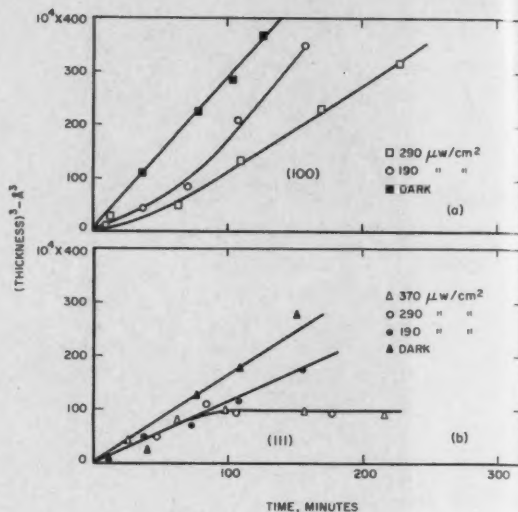
Many modern electronic devices, such as photocells, light flux meters, and solar radiation converters, depend on the well-known effect that light has on the movement of electrons in semiconductors. A recent NBS study by Jerome Kruger and Joan Calvert of the NBS Institute for Materials Research<sup>1, 2</sup> shows that during growth of semiconducting copper oxide films the movement of electrons creates a change in the charge on the positive copper ion which produces a retarding effect on the growth process. This investigation provides a better understanding of such processes and also of oxidation phenomena.

Other studies of the effects of light on  $\text{Cu}_2\text{O}$  have dealt with thick oxide films,<sup>3</sup> but because the trend in modern instrumentation is toward the use of thin films, this investigation was concerned with layers of less than 20 nanometers (200 Å).

*Boundary between dark and illuminated areas of copper single crystal surface oxidized in water at 25 °C. Upper region illuminated, lower kept dark. 500×*



Miss Joan Calvert carefully inserts a specimen into the apparatus to study the effect of light on the formation of oxide films on single crystal copper surfaces. Film thicknesses are measured with an ellipsometer (center bottom).



Cubic plot showing the effect of illumination of different intensities on the growth of oxide films on copper single crystals.

(a) (100) surface  
(b) (111) surface

Growth of the  $\text{Cu}_2\text{O}$  films on four crystallographic planes was studied and intensive analysis of the (100) and (111) planes was undertaken. These were the planes of fastest and slowest growth, respectively. The copper crystals were grown from 99.999+ percent pure copper, then immersed in a high-purity water bath for the film growth. Illumination was focused to a 0.5 cm diameter spot, and the light intensity was measured with a foot-candle meter. Ellipsometry was used to measure the thickness of the films.

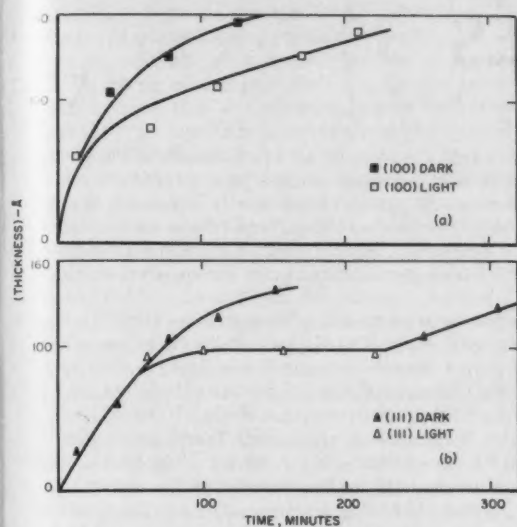
A control curve was obtained by growing a film in darkness and plotting the thickness as a function of time. The experiment was then duplicated except that light was focused on the growing film layer. It was found that the illumination slowed down the rate of film formation on both crystal faces studied, and the amount of retardation depended on the intensity of the illumination. This behavior can be explained on the basis of a competition between growth and dissolution reactions, the dissolution reaction being promoted by illumination.

When illumination is applied to the film surface, it causes electrons in the  $p$ -type  $\text{Cu}_2\text{O}$  semiconducting film

to migrate from the film into the metal. This means that some of the  $\text{Cu}^+$  ions in the  $\text{Cu}_2\text{O}$  are converted to  $\text{Cu}^{++}$  ions when the electrons leave and travel into the metal. As the cupric ( $\text{Cu}^{++}$ ) ions are more soluble in water than are  $\text{Cu}^+$  ions, dissolution increases and the layer decreases in thickness. The more intense the light the greater the flow of electrons, the end result being more dissolution reaction.

If the growth reaction is greater than the dissolution reaction, the film will grow as the difference in the magnitude of the two reactions. However, the rate of dissolution never becomes appreciably greater than the rate of growth because the growth rate is related to the thickness of the film. Thus, if the rate of dissolution surpasses the rate of growth, the film will immediately decrease in thickness until the growth rate equals the dissolution rate. The film thickness will then remain constant so long as the two reactions maintain equilibrium.

All other factors being the same, the amount of light applied to the growing surface is the only factor that affects the rate of dissolution. At constant light



Effect of illumination on the growth of oxide films on copper single crystals. The water bath in which the film was grown was in equilibrium with 99 percent helium and 1 percent oxygen gas mixture. Light intensity on the (100) surface (a) is approximately  $290 \mu\text{W}/\text{cm}^2$  and on the (111) surface (b) approximately  $270 \mu\text{W}/\text{cm}^2$ . Note the rise in the curve in plot (b) when the light was removed after 225 seconds.

intensity the rate of dissolution is constant. There is, however, a maximum number—a saturation value—of electrons that can be caused to flow from the film to the metal. For the (111) crystal face a maximum dissolution reaction was reached at about  $290 \mu\text{W}/\text{cm}^2$  of light intensity. When more intense light was applied the rate of film growth remained the same as at  $290 \mu\text{W}/\text{cm}^2$ . When the light was removed the rate of growth returned to the value observed in the darkness experiment.

This situation did not occur at the (100) face because the oxide film grows on this face at a greater rate than it does on the (111) face. In this case the rate of growth is always faster than the rate of dissolution.

It was discovered that when the water (in which the copper crystal was immersed) was in equilibrium with 100 percent oxygen, the application of light to the surface had very little effect on the growth rate, but when the bath was in equilibrium with an atmosphere containing 1 percent oxygen and 99 percent helium, illumination of the surface did exert a marked influence on the film formation process as described above.

This result is explained by the fact that when the partial pressure of oxygen is lowered,  $\text{Cu}_2\text{O}$  is the only oxide observed, whereas in an environment of 100 percent oxygen,  $\text{CuO}$ , which does not have the same photoconductive properties as  $\text{Cu}_2\text{O}$ , is formed as the outer part of the film, thus upsetting the growth-dissolution process.

No significant differences in oxidation behavior were observed when the wavelength of the radiating light was changed. However, experimental difficulties prevented the use of filters that exclude the long wavelength end of the absorption spectrum. It is in this range that illumination increases the photoconductivity; thus it cannot be conclusively stated at this time that the oxidation process is independent of wavelength. Future work will explore this question in greater detail.

<sup>1</sup> For further technical details, see Growth rate of oxide films on copper, NBS Tech. News Bull. 45, 134 (1961).

<sup>2</sup> The oxide films formed on copper single crystal surfaces in water, III. Effect of light, by J. Kruger and J. P. Calvert, J. Electrochem. Soc. 111, 1038 (1964).

<sup>3</sup> Retardation of copper corrosion by light, NBS Tech. News Bull. 42, 47 (1958).



## New Scale Established for LOW TEMPERATURES

A significant advance in low-temperature thermometry has been achieved at the NBS Institute for Basic Standards with the establishment of a scale of temperature for the region between 4 and 14 °K.<sup>1</sup> Based on the acoustical thermometer, this new absolute scale bridges the gap between the lower limit (10 °K) of the NBS 1955 Provisional Scale and the temperatures (2 to 5 °K) defined by the  $T_{58}$  Helium<sup>4</sup> Vapor Pressure Scale. As a result, NBS is now able to calibrate thermometers for industry at 1-degree intervals over the range from 2 to 20 °K, and the first such calibration has recently been completed.

Establishment of the new scale and calibration service is the result of a 9-year research program conducted by H. H. Plumb and G. Cataland of the Institute's low temperature laboratory.

Accurate measurement of temperatures below 20 °K is becoming increasingly important to the aerospace and other industries. Applications include the development of cryogenic fuels for rockets and missiles, work on solid-state devices for space-borne computers, design of high-field magnets, and studies of the phenomenon of superconductivity. However, until now the lack of an accurate scale providing continuous coverage of this temperature range has made it difficult to compare measurements made in one laboratory with those made in another.

For measuring temperatures below 20 °K, germanium resistance thermometers are commonly used. Those that are submitted for the new NBS calibration service are compared with standard germanium resistance thermometers of high sensitivity that have been calibrated with reference to the acoustical thermometer. This instrument makes use of the fact that the absolute temperature is proportional to the square of the speed of sound in an ideal gas (i.e., a gas at zero pressure). It consists essentially of a resonant (variable path, fixed-frequency sound wave) tube which is used to determine the wavelength and thus the speed. In practice, of

course, a real gas must be used and consideration given to the effect of pressure. However, the speed of sound is determined at pressures sufficiently low that a plot of speed versus pressure is linear and can be extrapolated to zero pressure. This procedure eliminates the need for pressure corrections and gives the speed in an ideal gas.<sup>2</sup>

Continuity in temperature measurement at NBS is obtained now from 2 °K to above 1336.15 °K by use of the  $T_{58}$  Helium<sup>4</sup> Vapor Pressure Scale (2 to 5 °K), the new NBS Provisional Scale of 2–20 (1965) (4 to 14 °K), the NBS 1955 Provisional Scale (10 to 90.18 °K and the International Practical Temperature Scale (90.18 °K to 1336.15 °K). Above 1336.15 °K, the temperature  $t$  is defined as the ratio of the spectral radiance of the blackbody at temperature  $t$  to the spectral radiance of a blackbody at the gold point (1336.15 °K).<sup>3, 4</sup>

Since the acoustical thermometer is an entirely new approach to precision thermometry, comparison of the scale based on this instrument with overlapping scales gives an independent check of the existing scales. Studies have shown that there is good agreement between the scale based on the acoustical thermometer and the NBS 1955 Provisional Scale in the 12 to 20 °K range, and the  $T_{58}$  Helium<sup>4</sup> Vapor Pressure Scale in the 2 to 5 °K range.

<sup>1</sup> An absolute temperature scale from 4 °K to 20 °K determined from measurements with an acoustical thermometer, by H. H. Plumb and G. Cataland, *J. Res. NBS* **69A** (Phys. & Chem.) No. 4 (July–Aug. 1965).

<sup>2</sup> Ultrasonic thermometer for low-temperature determinations, *NBS Tech. News Bull.* **47**, No. 1, p. 4 (1963).

<sup>3</sup> NBS temperature calibrations, *NBS Tech. News Bull.* **46**, No. 12, p. 186 (1962).

<sup>4</sup> The 1958 helium<sup>4</sup> scale of temperatures, introduction by F. G. Brickwedde; tables by H. van Dijk, M. Durieux, J. R. Clement, and J. K. Logan, *NBS Mono.* **10** (1960). For sale by the Superintendent of Documents, U.S. Government Printing Office, Washington, D.C., 20402, for 20¢.

H. H. Plumb (right) shows NBS Director A. V. Astin the first calibration report which the Bureau has issued for the calibration of a germanium resistance thermometer over the range from 2 to 20 °K.



Analysis of turbine-type flowmeters by the NBS Institute for Materials Research has defined the advantages and limitations of turbine flowmeters in cryogenic service. Results of a study<sup>1</sup> by W. J. Alspach and T. M. Flynn of the Institute's Cryogenics Laboratory staff indicate that, notwithstanding its limitations, the turbine-type flowmeter is one of the most practical types presently available.

Flow measurement of cryogenic fluids at the point of sale is the basis of customer billing. Flow measurement is also useful in ground and in-flight testing of rockets and missiles, and in cryogenic research laboratories it is essential. However, the very nature of cryogenic fluids makes them difficult to measure. They usually exist as saturated liquids, and thermal effects of the measuring device can be significant. The pressure drop through a flowmeter must be kept low to avoid superheating, and cryogenic fluids provide little or no lubrication for meter bearings.

The turbine-type volumetric flowmeter is probably the most popular of the various devices used to measure the flow of cryogenic liquids. It consists of a freely rotating blade supported by bearings inside a housing, and an electric transducer that senses the rotor speed. Rotor speed is a direct function of flow velocity which is translated into volumetric measurement. Popularity of the turbine-type flowmeter is due to its simple mechanical design, demonstrated repeatability—and the lack of simple direct meters for measuring mass flow.

Advantages of the turbine-type flowmeter are simplicity, small physical size, and less mass to cool. It has a flow range of from 5-to-1 to 10-to-1, and the signal output is linear, not a square-law function (as is the case with head-type meters). Response time is on the order of a few milliseconds, and the output is digital, making it readily adaptable to telemetry systems. No electric motor or other external driving force is necessary, as the fluid drives the rotor. No shaft or electrical seals are required between the fluid and the meter exterior, and should the rotor jam for any reason, there is no further impedance to flow of fluid.

# TURBINE-TYPE FLOWMETERS

*for cryogenic service*

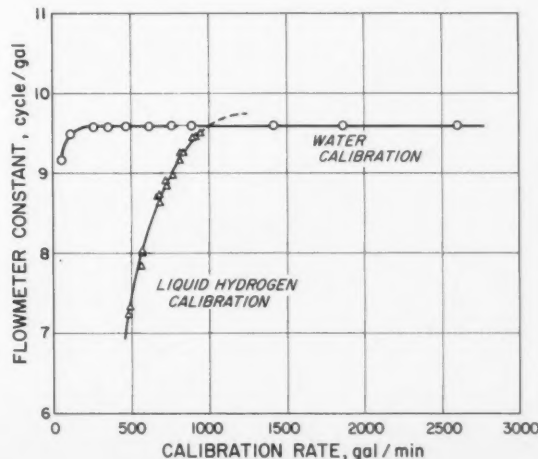
One disadvantage is the possibility of rotor overspeed. Unless they are supercooled, cryogenic fluids boil within transfer lines and thus exist in two phases—liquid and gas. The two-phase flow can be a "slug flow," with alternate slugs of liquid and gas; it may be "annular flow," with gas in the center and liquid around the circumference of the line; or, the mixture may be indiscriminate. Slug flow with intermittent high-velocity gas often overspeeds a rotor designed for low-speed fluid flow, and the heat generated by overspeeding can be dangerous in oxygen applications. Automatic devices to prevent overspeeding have generally proved less than completely satisfactory. Careful operator attention is often the best safeguard.

Turbine flowmeters are commonly calibrated with air or water. Several factors present in cryogenic applications, such as thermal effects and viscosity effects, cause variance from the air or water calibration.

In the use of turbine-type flowmeters, mounting configuration and upstream flow pattern are other factors to consider. Techniques for minimizing these and other causes of inaccurate readings are the objectives of research at NBS and other laboratories.

<sup>1</sup>For further information, see Considerations when using turbine-type flowmeters in cryogenic service, by W. J. Alspach and T. M. Flynn, *Advances in Cryogenic Engineering* 10 (in press).

*Variations between the liquid hydrogen calibration curve and that for water in an 8-in. volumetric flowmeter are caused by such factors as thermal and viscosity effects.*



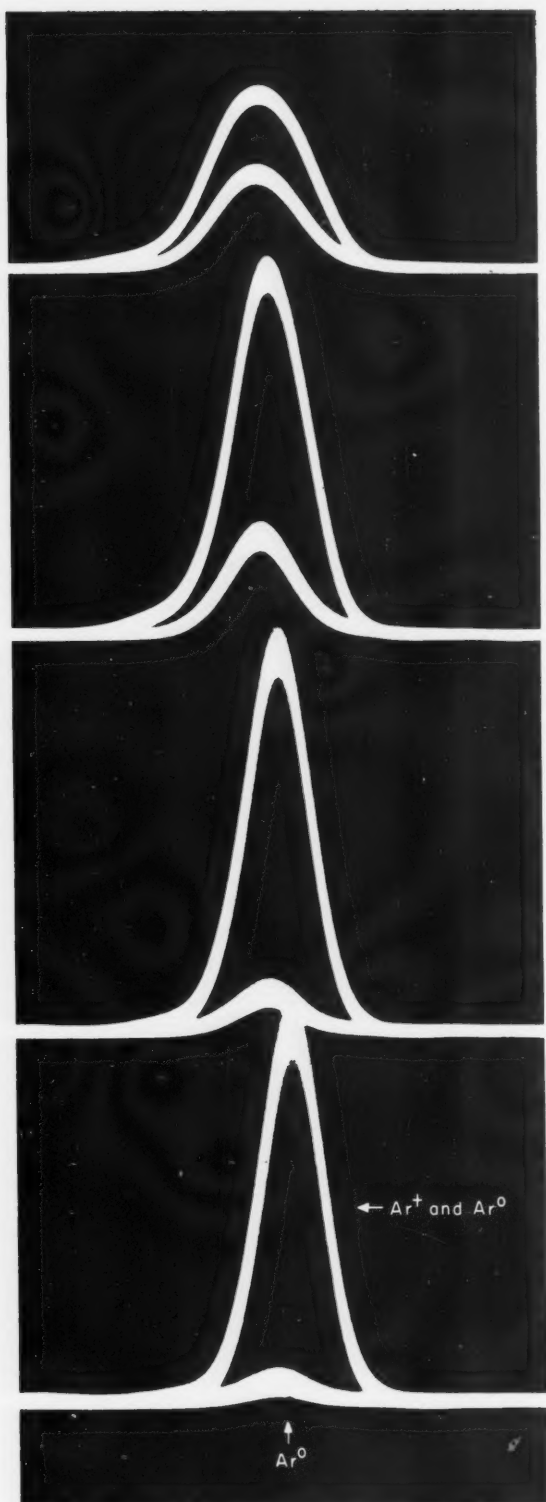
# IONIC COLLISION

A promising innovation, which may determine a new area of capability for the well-known time-of-flight (TOF) mass spectrometer, has been introduced at the Bureau.<sup>1</sup> R. E. Ferguson, K. E. McCulloh, and H. M. Rosenstock of the NBS Institute for Basic Standards have used a retarding potential applied at the end of the spectrometer drift tube, essentially converting the instrument into a two-stage TOF mass spectrometer. This modification and the favorable geometry of the ion path makes the instrument particularly useful for the study of a variety of elementary ionic processes, such as charge exchange, metastable transitions, and collision-induced dissociation.

In time-of-flight mass spectrometry, the charge-to-mass ratio of an ion is determined by measuring the time required for an ion of given kinetic energy (say 3 kV) to travel a known distance from an ion source to a detector. Since all ions are initially given the same energy, the heavier ions have longer travel times. The technique generally employed is to form ions in bunches, accelerate them rapidly to the desired kinetic energy, and let them travel through a long (1-meter) field-free drift region. The bunching is accomplished by successive application of a short ionizing electron pulse and an ion drawout pulse which starts the ions on their trip through the accelerating and drift regions. The start of the drawout pulse defines a zero time base relative to which the travel time is determined. At the end of the drift region the ion bunch, now separated into several groups in time according to mass, impinges on an ion multiplier and the signal is amplified. The full mass spectrum is displayed on an oscilloscope by triggering the horizontal sweep at the same time as the application of the drawout pulse, or at a predetermined later time.

By a simple modification of this arrangement, the NBS scientists were able to study ionic processes. A short region with a potential different from the drift tube is inserted at the end of the drift space. This, in effect, converts the apparatus into a two-stage time-of-flight mass spectrometer. The second stage serves to reveal any *changes* in the charge-to-mass ratio that a given ion may undergo in its travel through the field-free drift region.

As an extreme example, if some ions undergo charge exchange in the drift region, becoming fast neutral atoms or fragments, their travel times are unaffected



Oscilloscope showing the portion of the argon mass spectrum where the mass-to-charge ratio equals 40. The large peak on each double trace is the composite ( $Ar^+ + Ar^0$ ) and the small peak is the  $Ar^0$  component.



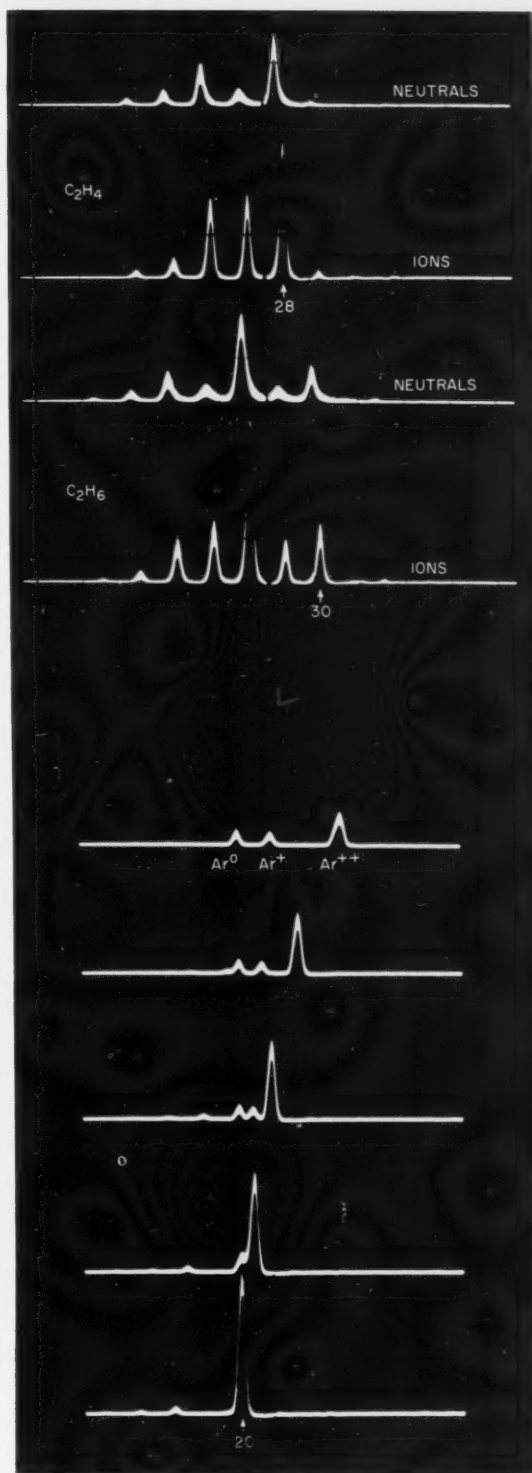
# ION PROCESSES

by the potential applied to the second analyzer, whereas all charged particles will be affected in known ways depending on the geometry, potential, and charge-to-mass ratio. Further, it is known that fast neutral fragments (e.g., 2–3 kV energy) can be detected by an ion multiplier. By varying the second stage potential it is thus possible to detect the formation of neutral fragments and to determine the mass-to-charge ratio of the ion from which they were formed.

In the present work the potential was applied to retard the ions. By applying sufficient voltage, a "neutral mass spectrum" is obtained consisting solely of neutral fragments formed by one or another process in the drift tube. The intensity relations between the two spectra are not straight-forward, reflecting the wide variety of process cross sections and detection efficiencies that may be encountered.

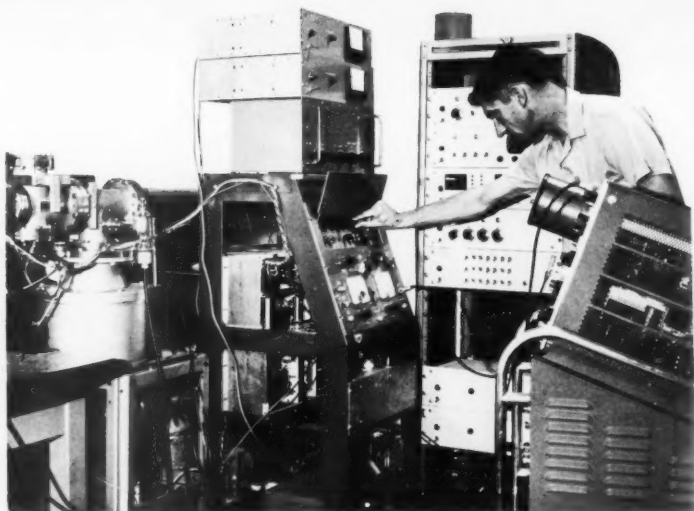
One class of processes that may be studied qualitatively by this technique is single and multiple charge transfer. For example, the oscillograph (bottom, right) shows the effect of various values of retarding potential on the region of the argon mass spectrum where the mass-to-charge ratio is equal to 20. The single peak without retardation is  $\text{Ar}^{++}$ . At higher values of the retarding potential this peak is split into three components. The stationary component consists of fast neutral argon atoms ( $\text{Ar}^0$ ) formed by collisions leading to two-electron charge transfer, the most voltage-sensitive one consists of that part of the  $\text{Ar}^{++}$  ion group which has not undergone any collisions, and the middle peak of moderate voltage sensitivity is  $\text{Ar}^+$  formed by single-electron charge transfer. Studies of pressure dependence indicate that only single collisions are involved.

Similar observations have been made on  $\text{N}_2^{++}$  and  $\text{C}_3\text{H}_6^{++}$ . In the latter cases it can be shown that there is single-electron charge transfer without subsequent decomposition of the ion. Without accurate knowledge of the comparative detection efficiencies of neutrals and ions, no quantitative data on cross sections



Oscillograph (above) of neutral atoms, resulting from collision processes, in the mass spectra of  $\text{C}_2\text{H}_4$  and  $\text{C}_2\text{H}_6$ . Oscillograph (below) of the peak in the argon mass spectrum corresponding to a mass-to-charge ratio of 20, showing charge transfer  $\text{Ar}^{++} \rightarrow \text{Ar}^+$  and  $\text{Ar}^{++} \rightarrow \text{Ar}^0$ . The bottom trace shows a typical time-of-flight mass spectrometer trace. The other traces (from bottom to top) show the effect of the application of a retarding potential (from 500 to 2000 volts in steps of 500 volts). Note that the individual mass peak components are almost unnoticeable at zero retarding potential, but quite distinguishable with sufficient retarding potential applied. Thus, the individual components of any given mass peak may be visually represented and easily studied.

K. E. McCulloh adjusts the electron beam current in the time-of-flight mass spectrometer. Spectrometer tube is seen in background (left).



can be obtained as yet. However, relative cross sections for various target gases could be determined with good accuracy.

Another class of processes which can be studied by this technique is spontaneous unimolecular ion decompositions. These processes are well-known in magnetic deflection mass spectrometry under the term "metastable transitions." In the electron impact ionization and dissociation of butane two of these processes are:  $C_4H_{10}^+ \rightarrow C_3H_6^+ + CH_4$  and  $C_4H_{10}^+ \rightarrow C_3H_7^+ + CH_3$ .

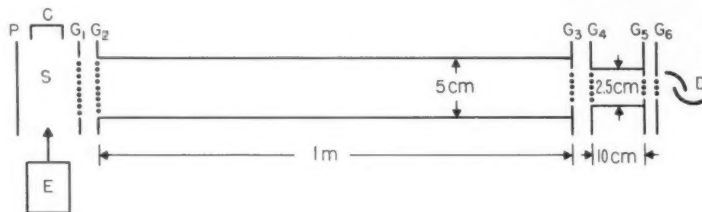
In a magnetic mass spectrometer such processes, when they occur after acceleration, but before magnetic deflection, appear as diffuse peaks at non-integral mass numbers determined by the relation

$$M_{\text{metastable}} = \frac{M_{\text{product ion}}^2}{M_{\text{reactant ion}}}$$

and the process is inferred from knowledge of this mass number.

In a time-of-flight apparatus the charged and neutral products of decomposition anywhere in the drift region will travel along with the reactant ion group. However, second-stage retardation can again be used to locate the neutrals and to determine the mass of the product ions. These product ions are far more retardation-sensitive than the normal ions because of their reduced momentum and kinetic energy. In the present technique the abundance of these processes is significantly larger (roughly a factor of 3 to 10) because of the increased length of the drift space compared to a magnetic deflection mass spectrometer. More important, by varying the second-stage retardation voltage the mass number of the reactant and product ions can be determined directly.

<sup>1</sup> For further technical details see, Observation of the products of ionic collision processes and ion decomposition in a linear, pulsed time-of-flight mass spectrometer, R. E. Ferguson, K. E. McCulloh, and H. M. Rosenstock *J. Chem. Phys.* **42**, 100 (1965).



Schematic of a conventional time-of-flight mass spectrometer is shown with the retarding potential device. Gas is admitted to the source area (S) and ions are formed as the beam of electrons from the gun (E) passes through the gas. The backing plate (P) is operated at ground potential and the electrons are collected at (C). The newly formed ions are pulled out by the accelerating grid ( $G_1$ ,  $G_2$ ) and then enter the one meter drift tube. Grid ( $G_3$ ) is at drift tube potential, and the decelerating grids are at variable potential ( $G_4$ ,  $G_5$ ) and first dynode potential ( $G_6$ ). The NBS modification which permits the study of elementary processes, is attached to the end of the drift tube just before the first stage (D) of the electron multiplier. Spacing between the grids is about 3.5 mm.

## Colloidal Data

### Contracts Let

Two contracts for the compilation of data in colloid chemistry have been let by the NBS Office of Standard Reference Data, which administers the National Standard Reference Data System. The data compilations are part of a broad program in the general field of colloid and surface properties. This program has been undertaken as a joint effort by NBS and the National Academy of Science-National Research Council Committee on Colloid Chemistry.

One contract, with the University of Southern California at Los Angeles, provides for the collection and evaluation of data on critical micelle concentrations in colloidal systems. The other is with the Clarkson College of Technology at Potsdam, N.Y., and involves the collection and evaluation of data on light scattering in colloidal systems. Both compilations will be kept up to date as new data become available. The data will be useful in a wide variety of scientific and technological fields, including basic studies of the physical structure of colloidal systems, studies of the sizes and shapes of colloidal aggregates, methods of control of air and water pollution, and studies of detergency.

The National Standard Reference Data System (NSRDS) is the outcome of action taken by the President's Office of Science and Technology to provide standard reference data needed for scientific, techno-

logical, and economic advancement.<sup>1</sup> "Standard reference data" means critically evaluated quantitative information relating to a property of a definable substance or system. Tabulations of such data are of great value in the design of an experiment or of equipment, but they are often hard to find or out of date. The purpose of the NSRDS is to provide a storehouse of up-to-date tabulations that will be readily accessible for user needs. This end is to be achieved through a broad-based, comprehensive effort by scientists both in and outside government.

NBS has now identified priority areas for NSRDS compilations with the assistance of specialists on various advisory panels. These areas cover: (1) nuclear data; (2) atomic and molecular data; (3) solid state data; (4) thermodynamic and transport properties; (5) chemical kinetics; (6) colloid and surface properties; and (7) the mechanical properties of materials. The data to be furnished by the University of Southern California and the Clarkson College of Technology fall within the sixth category. Data compilation projects are now under way at NBS laboratories in several of the categories, and contracts are being negotiated with other organizations for similar activities.

<sup>1</sup> For details, see National Standard Reference Data System Plan of Operation, NSRDS-NBS 1, December 1964, by Edward L. Brady and Merrill B. Wallenstein, available from the Superintendent of Documents, U.S. Government Printing Office, Washington, D.C., 20402 (15¢); also, National Standard Reference Data System, NBS Tech. News Bull. 47, 138 (1963).

## New Standard for Hall Devices

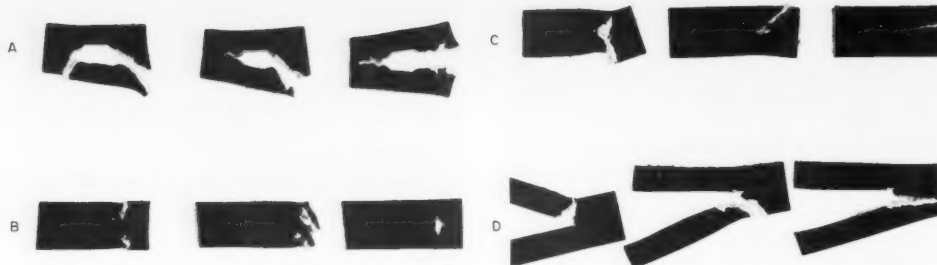
A new standard developed by the NBS Institute for Applied Technology gives nomenclature and symbols for describing Hall effect devices, as well as measurement procedures for determining some of their characteristics. The standard was developed by Sherwin Rubin of NBS for the Bureau of Naval Weapons and has been adopted by the Navy as a first step in the preparation of a tri-service standard. The standard MIL-STD-793-1(WP)<sup>1</sup> is the first in a series designed to serve as a procurement document for Hall effect devices.

Although designed primarily to facilitate naval procurement, the specification was coordinated with representatives of industry so that it would be of maximum utility to government, industry, and university laboratories. It is thus compatible with prevailing practices among nonmilitary groups.

In the Hall effect, an electric current flowing at right angles to a magnetic field generates a voltage gradient perpendicular to both current and field. For many years a laboratory curiosity, this phenomenon is now widely utilized in semiconductors and other electronic devices. Applications of the effect range from modulating single sideband radio transmissions to measuring nuclear power, and its suitability is unique in measuring power and magnetic field strength and in performing arithmetic operations on analog quantities.

With increasing use of Hall effect devices, diverse systems of terminology and characterization have developed which confuse users and make procurement difficult. The new specification was developed to solve these problems.

<sup>1</sup> MIL-STD-793-1(WP), issued 15 December 1964, is available from the Bureau of Naval Weapons (RREN-5), Department of the Navy, Washington, D.C., 20360.



# NEW TEST

## Slit-Tear Test

Leather strength tests are important to industrial users of leather and are relied upon extensively for the selection of military footwear. A comparison of the slit-tear test<sup>1</sup> for the strength of leather, a new test approved by the International Union of Leather Chemists' Societies, with three other leather strength tests already described in the Federal Specifications has been carried out by Thomas J. Carter of the NBS Institute for Materials Research. The results showed good correlation of the tearing load for the new test with the average tearing load for all tests.<sup>2</sup>

The three tests mentioned in the Federal Specifications which measure the tear resistance of leather are the double-hole stitch-tear (or buckle tear) test, the single-hole stitch-tear test, and the tongue-tear test. Each represents a type of tear that may occur in certain similar applications.

The double-hole stitch-tear test is used to simulate the behavior of stitched seams in shoes, and it is also used in specifications for glove and garment leather. Two holes are punched in one end of a small (1 in. by 2 in.) rectangular specimen. The ends of a U-shaped wire are passed through the holes and clamped in one jaw of the testing machine, and the free end of the specimen is clamped in the other jaw which is movable.

The single-hole stitch-tear and tongue-tear tests are designed to test heavy leather, but may also be used satisfactorily to compare the strength of different types of light leathers. The single-hole stitch-tear test is performed in a manner similar to that of the double-hole stitch-tear test except that the leather specimen has only one hole.

For the tongue-tear test, a hole is punched 1 inch from the end of a (1 in. by 6 in.) leather piece which is split into two strips, or tongues, along the centerline to the hole. The specimen is mounted by clamping one strip in the fixed jaw and the other strip in the movable jaw of the testing machine.

For the new slit-tear test, a longitudinal slit is punched in the center of the leather specimen. Two L-shaped metal pieces are used to hold the specimen for testing. Both flanges pass through the slit from the same side of the specimen; the web of each piece is used to clamp the specimen into the testing machine.

Because the concept of strength is not defined in an absolute way, but only in terms of the tests to be evaluated, the sensitivity criterion<sup>3</sup> was used as the basis for comparison of the four methods. Results of the slit-tear test showed good correlation with the average tearing strength for all tests.

The principal advantage of the slit-tear test is that it can be performed more rapidly than the other tests. In addition, the equipment used to apply the load does not come into direct contact with the fibers at the point of tear as it does in the double-hole stitch-tear and single-hole stitch-tear tests where the fibers may be subjected to a cutting action as well as to the tearing forces.

Because of the reduced time necessary to perform the slit-tear test and because this test gives results in good correlation with the average tearing strength of the other three tests already described in the Federal Specifications, it is expected that the new slit-tear test will become as widely used as the other three tests.

<sup>1</sup> E. Baumann, *Das Leder*, **8**, 196 (1956).

<sup>2</sup> For further details, see Comparison of tearing strength tests for leather, by Thomas J. Carter, *J. Am. Leather Chem. Association* **LX**, No. 1, 4-14 (January 1965).

<sup>3</sup> Sensitivity—a criterion for the comparison of methods of test, by John Mandel, and R. D. Stiehler, *J. Res. NBS* **53**, 155 (September 1954).

above:

Ruptured specimens from four leather strength tests carried out at the Bureau. The new slit-tear test (A), was compared with the double-hole stitch-tear test (B), the single-hole stitch-tear test (C), and the tongue-tear test (D). The direction of the tear is not dependent entirely on the direction of the applied force but follows the path of lowest fiber resistance.

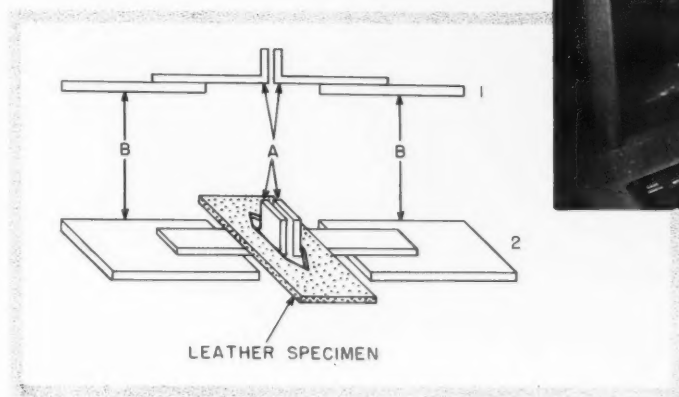
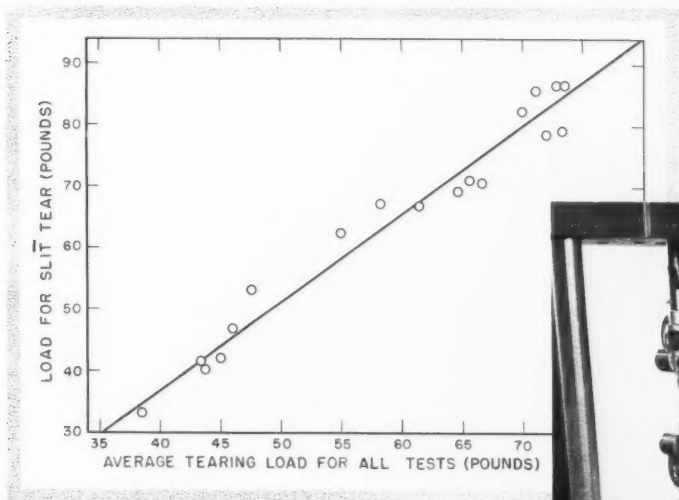
Experimental values of the tearing load for the slit-tear test shown plotted against the tearing load average of this test with three other leather strength tests already described in the Federal Specifications.

Thomas J. Carter adjusts a leather sample in preparation for a slit-tear test of the strength of the leather.

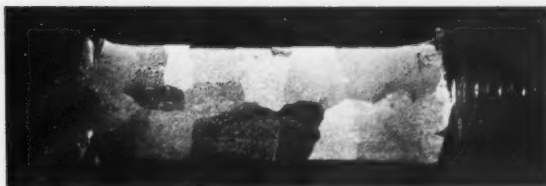
(1) Side view of specimen holder, (2) front view with specimen mounted, (A) L-shaped metal pieces which hold the specimen, (B) support bars.

# WEAR STRENGTH TEST FOR LEATHER

## Slit-To Test Compared with Three Other Strength Tests







## Aluminum Strain Behavior Studied

Recent work at the NBS Institute for Materials Research sheds some light on the behavior of residual strains observed in metals after plastic deformation. These strains exhibit an isotropic pattern, even though the metal itself is anisotropic. X-ray measurements on aluminum now indicate that individual grains in the crystal lattice of the metal may be constrained by their neighbors in such a way that the strains are forced into the pattern.<sup>1</sup> These findings should prove useful in basic studies on metal fatigue and on the mechanical properties of metals.

The residual strain patterns induced in metals have been extensively studied because of their influence on metal strength and elasticity. There are at least two possible explanations for the isotropic nature of these patterns. One is the "constraint" concept in which softer regions in a stressed metal are acted upon by randomly oriented neighboring grains and by hard material at grain or subgrain boundaries. The other is an "averaging" concept in which the strains—coming typically from hundreds of crystallites—represent a nonzero average that exhibits isotropy.

Scientist C. J. Newton of the Institute's metallurgy staff undertook the present work to obtain a better understanding of the phenomenon. He utilized the shift of x-ray diffraction peaks as an indication of strain in individual coarse grains of a polycrystalline aluminum. In such grains the ratio of the softer regions to the harder regions is about a hundred thousand times larger than it is in fine-grained aluminum. Furthermore, since the x-ray diffraction peak position is determined by the softer material, the peak shifts observed in the study represent the elastic strain and the related stress in the soft part of the material only.

The specimen used in the experiments was specified by the supplier to be 99.99 percent pure aluminum. It had threaded ends and a reduced section about 1 1/4 in. long, with a square cross section 1/2 in. on a side. It was in a fully annealed, stress-free condition with

etched surfaces and with grains ranging in diameter from about 1/16 to 1/4 in.

The crystallographic orientation of 51 grains on two faces of the reduced section were determined by back-reflection Laue patterns. An x-ray diffractometer method was then used, with Cu characteristic radiation, to determine the diffraction peak positions from the (511) and (333) planes.

Subsequently the specimen was uniaxially strained in tension to a final plastic strain of 10 percent. It was then realigned in the diffractometer and the peak positions of the 50 grains were redetermined. Values were found for 229 different strains. The observed shifts in peak position were small, but they were generally several times the estimated uncertainty of the measurement. Both positive and negative peak shifts were found, some amounting to more than 0.10 degree.

A consistent set of three principal strains related directly to the geometry of the specimen and to its axis of deformation could not be deduced from the observed strains. Thus an isotropic pattern was not induced in this coarse-grained material. Nor was any self-consistency or general trend or "average" behavior anywhere in evidence among the observed strains in the grains.

It would therefore seem that, in view of the complete absence of any trace of consistent trend, the averaging concept is less likely than the constraints explanation for the isotropic behavior observed in fine-grained material. The lack of isotropic behavior found in the present study can be attributed to the lessening of the influence of neighboring grain constraints and to the larger relative amounts of soft regions in the coarse-grained than in fine-grained aluminum. The greater distances over which constraining forces would be required to act in the present instance would account for the marked difference between the observed strain behavior and that found in ordinary fine-grained materials.

*One of the surfaces of the specimen, before (left) and after (right) plastic deformation. The right photo shows considerably inhomogeneity of strain, that is, the specimen did not elongate uniformly, as is the case with fine-grained materials.*

<sup>1</sup> For further details, see X-ray measurement of residual strains in individual grains of polycrystalline aluminum, by Clarence J. Newton, J. Research NBS 68C (Eng. and Instr.), 249 (1964).

Clarence J. Newton fits the polycrystalline aluminum specimen to a specially designed goniometer specimen holder.



THE FIRST calcium aluminate sulfates ever prepared free of carbon dioxide were recently supplied to a committee of the Highway Research Board (HRB)\* by the NBS Institute for Applied Technology. These compounds,<sup>1</sup> prepared by H. A. Berman of the Institute staff, should prove valuable to research laboratories engaged in the HRB committee's cooperative program for studying the properties of portland cement constituents. They should also aid in improving methods of identifying the compounds found in portland cement.

The two compounds, calcium aluminate monosulfate ( $3\text{CaO} \cdot \text{Al}_2\text{O}_3 \cdot \text{CaSO}_4 \cdot 12\text{H}_2\text{O}$ ) and calcium aluminate trisulfate ( $3\text{CaO} \cdot \text{Al}_2\text{O}_3 \cdot \text{CaSO}_4 \cdot 32\text{H}_2\text{O}$ ), play a significant role in controlling the setting rate of portland cement. They also occur when concrete is destroyed by exposure to sea water or sulfate-containing waters.

The HRB committee has circulated these and other relatively pure materials to certain research laboratories here and in other countries, where various techniques will be applied to study the structure and properties of the compounds. When portland cement is mixed with water, it sets to a hard mass. A better understanding of the many reactions that take place during the setting period, and of the many hydration products formed, is needed to improve the strength and durability of concrete.

Although purity is not a characteristic of the chemical compounds when they appear in cement, it is essential to study them in a form as pure as possible. Carbon dioxide is one of the impurities most difficult to avoid. This gas, present almost everywhere, forms carbonates which look and act like the calcium aluminate sulfates and enter into solid solutions with them, changing such fundamental data as planar atomic spacings, unit cell dimensions, and refractive indices. Normal laboratory precautions in the past, at NBS and elsewhere, have managed to keep the  $\text{CO}_2$  content of

the calcium aluminate sulfates as low as 0.2 percent, occasionally 0.1 percent, but no lower. The materials supplied to the HRB committee by NBS, however, were produced with a  $\text{CO}_2$  content of 0.0 percent, and possibly 0.00 percent for the monosulfates.<sup>2</sup>

The apparatus designed and used to eliminate the  $\text{CO}_2$  consists of three units: a closed precipitation system, a closed filtration system, and a glove box enclosure for storage and conditioning of the materials, in which the atmosphere is kept  $\text{CO}_2$ -free, and an air-lock permits transfer of materials into and out of the box without contamination of the air inside the box. The basic problem in the precipitation of the monosulfate was to bring together large quantities of dilute solutions (200 liters of calcium hydroxide and about 2 liters of aluminum sulfate) within a short time and to keep them  $\text{CO}_2$ -free. With only 58-liter bottles available for storing the solutions and mixing the reactants, it was necessary to mix four batches of reactants and to allow the precipitate to accumulate in the reaction bottle, meanwhile drawing off the spent liquor between batches. The process had to be performed quickly inasmuch as the monosulfate is unstable and, if left in solution, changes after about seven hours to the trisulfate. Moving large quantities of liquid rapidly requires rapid movement of equal volumes of air, but the effective removal of  $\text{CO}_2$  from the air requires slow passage through  $\text{CO}_2$ -absorbents. This contradiction was solved by preparing the  $\text{CO}_2$ -free air slowly, trapping it in 50-liter pockets, and moving the trapped pockets of air back and forth as the solutions displaced them by gravity in a closed system. This principle was followed in preparing the solutions (directly in the original reagent bottles in the case of the aluminum sulfate), mixing them, drawing off spent liquor, and filtering them. (Continued on next page)

## First cement compounds free of $\text{CO}_2$



Calcium aluminate sulfates are dried to a constant weight in a desiccator within a glove box containing a  $\text{CO}_2$ -free atmosphere. H. A. Berman is shown reaching into the glove box.

# CLEARINGHOUSE

FOR FEDERAL SCIENTIFIC AND TECHNICAL INFORMATION

## New Guide to Federal Research and Development Reports

A single, easy-to-use guide to new unclassified Government-sponsored scientific and technical reports began publication at the Bureau in June. The new *Government-Wide Index to Federal Research and Development Reports* is prepared by the Bureau's Clearinghouse for Federal Scientific and Technical Information in cooperation with the Department of Defense, National Aeronautics and Space Administration, and the Atomic Energy Commission.

The Index is published on the 15th of each month and covers new documents listed in the four major technical report announcement journals issued by the cooperating agencies for the prior month—U.S. Government Research and Development Reports (USGRDR), Technical Abstract Bulletin (TAB), Scientific and Technical Aerospace Reports (STAR), and Nuclear Science Abstracts (NSA). It will provide scientists, engineers, and research managers with a single source to this Federal technical information.

The Index is prepared by computer manipulation of data records prepared by the four agencies for other purposes. These records are reformatted by the Clearinghouse to meet the requirements of the five indexes that make up the publication—subject, personal author, corporate source, report number, and accession number. Initially, most of the computer programming and processing is being performed for the Clearinghouse by the Aerospace Research Applications Center (ARAC) at Indiana University.

The Index is now being reproduced from the computer printout furnished by ARAC, but by late summer

it will appear in conventional printing format. A typical entry is shown below:

The access points to technical reports listed in Government-Wide Index include:

- Subject
- Personal Author
- Corporate Source
- Report Number
- Accession Number

Each index is arranged alphanumerically (that is, alphabetic data precedes numeric data). A typical entry is as follows:

Index Entry	Title of Report	Volume No.	Issue No.
TITANIUM ALLOYS SOLID SOLUTION HARDENING OF ALPHA AND BETA TITANIUM PB-165 379		1.00	0.50
Accession/Report No.*	Hard Copy Price	Microfiche Price	Journal Code**

\*The Accession/Report number is the number used by the announcing agency to uniquely identify the report; this number should be used in all correspondence with that agency.

\*\*The code for the journal volumes and issues in which the reports are announced and abstracted are as follows:

- U—U.S. Government Research and Development Reports, Clearinghouse for Federal Scientific and Technical Information.
- T—Technical Abstract Bulletin, Defense Documentation Center of the Department of Defense.
- N—Nuclear Science Abstracts, Atomic Energy Commission.
- S—Scientific and Technical Aerospace Reports, National Aeronautics and Space Administration.

The Government-Wide Index to Federal Research and Development Reports is sold by the Superintendent of Documents, U.S. Government Printing Office, Washington, D.C., 20402. The price is \$10 for a one-year subscription (12 issues); add \$2.50 for foreign mailing; single issue \$1.25. Remittances should be made payable to the Superintendent of Documents.

## Cement Compounds—Continued

The filtration medium was a large sintered-glass funnel, equipped with an oversize rubber stopper specially made at NBS, and capable of being closed off from the laboratory air both at the inlet and outlet. The entire sealed assembly of filter funnel and filtered precipitate was transferred to a glove box containing a CO<sub>2</sub>-free atmosphere, so that the material never came in contact with the laboratory air. In the glove box, the precipitate was transferred to a desiccator containing crystals and saturated solution of MgCl<sub>2</sub>·6H<sub>2</sub>O, in which an atmosphere of 33 percent relative humidity can be maintained. The precipitate was dried under these conditions to constant weight, the hydrates formed thereby containing the quantity of H<sub>2</sub>O indicated in the formulas.

Equally as essential as preparing the materials in a CO<sub>2</sub>-free atmosphere was the necessity of keeping them uncontaminated by CO<sub>2</sub> during shipment to the cooperating research laboratories. Still inside the glove box, the samples were packaged in glass vials. The entire vial was then sealed in a glass tube containing calcium oxide as a carbon dioxide absorbent.

\*HRB Task Group MCB-6a, project Committee MCB-6, on Cooperative Determination of Relative Effectiveness of Various Analytical Methods in Identifying the Phases in Hydrated Cement Pastes.

<sup>1</sup>For further information, see Preparation of a carbonate-free complex calcium aluminate, H. A. Berman, J. Research NBS 69A (Phys. and Chem.) No. 1, 45-51 (Jan.-Feb. 1965).

<sup>2</sup>No carbon dioxide was found in any of the monosulfate samples, but the limit of sensitivity in the analysis is estimated to be 0.03 percent. The trisulfate contained 0.04 percent CO<sub>2</sub>.



# Extended Study of Denture Fit

## *Loss of Fit Caused by Tissue Changes*

Dentures and bridges are worn by nearly one-half of the American population over 21. A 5-year study on the fit of complete dentures, recently completed at the NBS Institute for Materials Research, showed that the gradual deterioration of fit was caused by changes in the patterns of contact of the mouth tissues with the dentures and not by dimensional changes in the dentures. These results indicate that the widely held belief that dentures change dimensionally in service is not correct.<sup>1</sup>

The work was conducted by Julian B. Woelfel and George C. Paffenbarger, American Dental Association Research Associates at the Bureau, and William T. Sweeney of the NBS Institute for Materials Research.

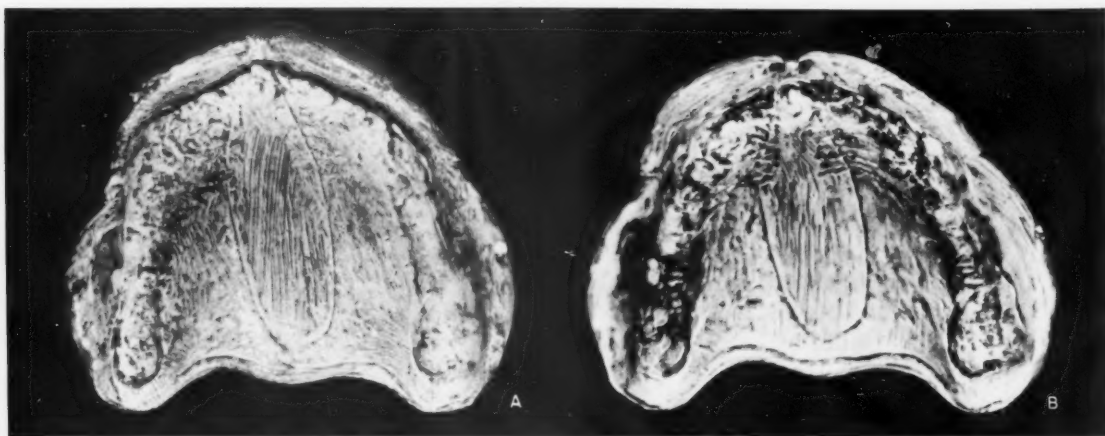
After establishment of a proper initial fit of a denture, the fit was checked several times each year by the dentist. Each patient was also asked for his personal evaluation. These observations were supplemented by concurrent measurements of the dimensional changes of the dentures caused by processing procedures and by use.

Laboratory tests of 200 dentures showed that the largest dimensional change of any of the dentures during processing, or during use, was not large enough for either the patient or the dentist to detect any impairment in fit or stability. However, a change in the size and shape of the mouth tissues that support the dentures was apparent during several years of use. This change in the tissues caused a decrease in retention and stability, as well as in the alignment of the upper and lower dentures relative to each other. A misalignment of upper and lower dentures causes a high degree of unsteadiness and very poor retention.

Changes in the denture-supporting tissues were determined by use of pressure-indicator-paste patterns. In this method, a thin layer of opaque paste is painted on the tissue-bearing surface of the denture, which is then carefully seated in the patient's mouth. The paste pattern, obtained after the patient simulates chewing for a few minutes, shows the areas of hard contact with the tissues.

*Pressure-indicator-paste patterns of an upper denture. Pattern A was taken in July 1958, at the time the denture was inserted. Pattern B, taken three years later in July 1961, shows large and heavy contact areas not present in Pattern A. The denture*

*remained stable in dimensions during these three years as the total molar-to-molar change was only -0.14 percent. Areas in hard contact appear dark.*





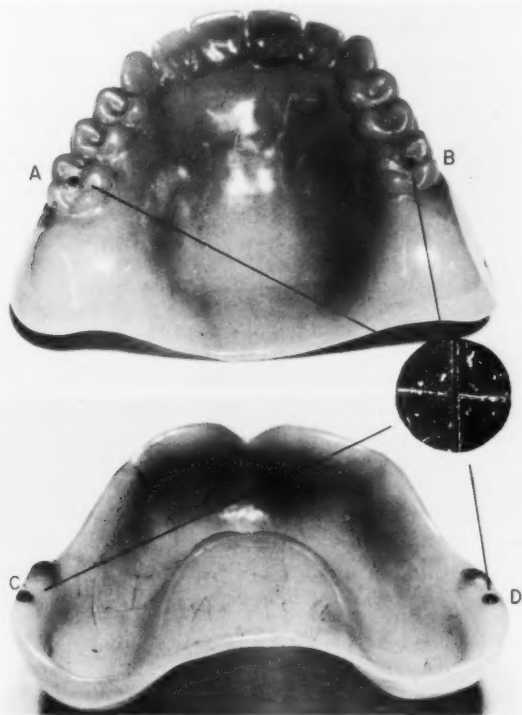
## NBS Issues

### Radioactivity Standards

The relative continued effectiveness of a properly made denture depends on how well the tissues of the wearer tolerate the stress imposed by the denture. With few exceptions, a denture needs to be relined within 3 years because of changes in the supporting tissue. The importance, to denture comfort, of uniform contact between mouth tissues and the denture was demonstrated in a test in which a purposely warped denture was made to function satisfactorily and fit comfortably by selectively grinding the areas in hard contact with the tissues (determined from pressure-indicator-paste patterns).

<sup>1</sup>Clinical evaluation of complete dentures made of eleven different types of denture base materials, by Julian B. Woelfel, George G. Paffenbarger, and William T. Sweeney, *J. Am. Dental Association* (in press).

Dimensional changes between the last molars (AB) and the flanges (CD) were measured with a microscope. The enlargement (right) shows fine cross hairs on stainless steel pins inserted at A, B, C, and D.



Of the more than 500 standard reference materials issued by the NBS Office of Standard Reference Materials, over 40 are radioactivity standards. Seven new or renewed radioactivity standards have recently been announced and are now available: <sup>1</sup> Sodium 22, iron 55, zinc 65, yttrium 88, cesium 137-barium 137m, cerium 141, and americium 241.

NBS Standard No. 4904-A, americium 241, which emits  $\alpha$  particles and  $\gamma$  rays, is for calibrating instruments used in civil defense, such as  $\alpha$ -particle survey meters. This standard is also used to calibrate instruments used to study the effects of  $\alpha$ -emitting nuclides on living organisms. Long half-life and source stability makes this standard usable for long periods of time.

NBS Standard No. 4991-B, sodium 22, emits positrons and 1.28-MeV  $\gamma$  rays, and is used for calibration of  $\gamma$ -ray spectrometers and for instruments employed in positron studies.

NBS Standard No. 4929-B, iron 55, emits 5.9-keV x rays and is issued to calibrate instruments that monitor iron 55, a tracer used in both biological and industrial investigations.

NBS Standard No. 4992-B, zinc 65, which emits x rays and  $\gamma$  rays (1.114 MeV) is issued mainly for calibrating  $\gamma$ -ray spectrometers.

NBS Standard No. 4998, yttrium 88, primarily used for calibrating both x-ray and  $\gamma$ -ray spectrometers emits 14.1-keV x rays, and 0.908-MeV, 1.853-MeV, and 2.762-MeV  $\gamma$  rays.

NBS Standard No. 4200, cesium 137-barium 137m, is a  $\gamma$ -ray point-source standard and was developed in response to the needs of a large number of laboratories involved in counting cesium 137 in humans (whole-body counting). Cesium 137 occurs in fallout and eventually finds its way into the human body.

NBS Standard No. 4946, cerium 141, is a short-lived radionuclide issued to calibrate  $\gamma$ -ray and  $\beta$ -ray detectors. This standard is of interest to scientists measuring fission products that occur in fallout.

Five other standards are currently out of stock. These are standards of radionuclides which have relatively short half-lives and the maintenance of a permanent stock is not feasible. Two of these, iodine 125 and mercury 203 (NBS Nos. 4944-B and 4932-D) will be reissued in the near future. The other three, scandium 46, strontium 85, and niobium 95 (NBS Nos. 4939, 4938, and 4937) may be available within a year. Inquiries about these standards will be filed for notification of reissues.

Chemically stable solutions of many of the radionuclides previously standardized by NBS may be submitted for calibration. Details of calibration services may be obtained from the Radioactivity Section of the National Bureau of Standards.

<sup>1</sup> Standard reference materials may be ordered from the Office of Standard Reference Materials, National Bureau of Standards, Washington, D.C., 20234.



# Cement Study Reveals New Compound

During an investigation of hydraulic cements in the Building Research Division of the NBS Institute for Applied Technology, a hitherto unreported crystalline compound was detected. Further investigation<sup>1</sup> of this compound by Institute staff member E. T. Carlson revealed it to be a gehlenite hydrate with the formula  $2\text{CaO} \cdot \text{Al}_2\text{O}_3 \cdot \text{SiO}_2 \cdot \text{H}_2\text{O}$ . Inasmuch as the hydrate was formed only at temperatures higher than 250 °C, it would not be expected in steam-cured concrete made under current practice. It should, however, be of interest to anyone investigating hydrothermal treatment of cements or blast furnace slags.

From the x-ray diffraction pattern it was deduced that the compound has a body-centered cubic structure, with unit cell edge 0.883 nanometer (8.83 Å). The index of refraction is 1.628. The formula,  $2\text{CaO} \cdot \text{Al}_2\text{O}_3 \cdot \text{SiO}_2 \cdot \text{H}_2\text{O}$ , indicates a relationship to the mineral gehlenite,  $2\text{CaO} \cdot \text{Al}_2\text{O}_3 \cdot \text{SiO}_2$ . This fact is of interest because gehlenite has been reported to react with water, under hydrothermal conditions, to form a hydrogarnet, in which the ratio of CaO to  $\text{Al}_2\text{O}_3$  is 3 to 1 rather than 2 to 1. At room temperature, gehlenite is practically inert toward water, but a compound of composition  $2\text{CaO} \cdot \text{Al}_2\text{O}_3 \cdot \text{SiO}_2 \cdot 8\text{H}_2\text{O}$ , often called gehlenite hydrate, can be prepared by hydration of glasses of gehlenite composition as well as by other means.

In the present work a preparation of synthetic gehlenite was converted hydrothermally to the new hydrate,  $2\text{CaO} \cdot \text{Al}_2\text{O}_3 \cdot \text{SiO}_2 \cdot \text{H}_2\text{O}$ , at 260 to 650 °C, but at a lower temperature (182 °C) a hydrogarnet was formed. Similar results were obtained with a specimen of natural gehlenite, but with minor differences attributed to impurities ( $\text{MgO}$  and  $\text{Fe}_2\text{O}_3$ ) in the natural mineral.

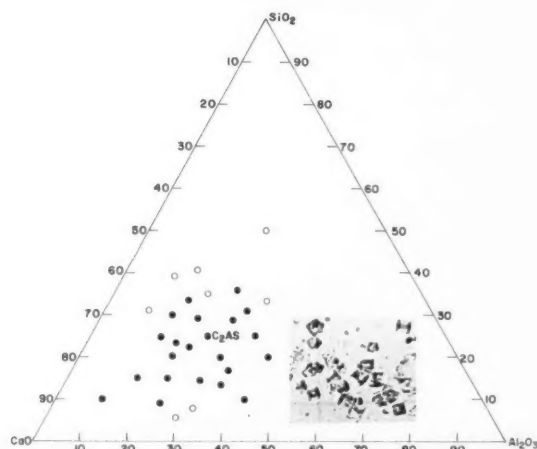
<sup>1</sup>For further information, see Hydrothermal preparation of a gehlenite hydrate, by Elmer T. Carlson, *J. Res. NBS* 68A (Phys. and Chem.) No. 5, pp. 449-452 (Sept.-Oct. 1964).

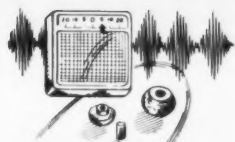
Above: E. T. Carlson checks the temperature on the furnace used to treat hydraulic cements hydrothermally at NBS.

The previously mentioned gehlenite hydrate,  $2\text{CaO} \cdot \text{Al}_2\text{O}_3 \cdot \text{SiO}_2 \cdot 8\text{H}_2\text{O}$ , reacted hydrothermally in a similar manner, forming the monohydrate at the higher temperatures, but in the intermediate range converting to a hydrogarnet. It is concluded, therefore, that on hydrothermal heating,  $2\text{CaO} \cdot \text{Al}_2\text{O}_3 \cdot \text{SiO}_2 \cdot 8\text{H}_2\text{O}$  is first converted to a hydrogarnet, with release of part of the  $\text{Al}_2\text{O}_3$  and  $\text{SiO}_2$ . Then, as the temperature is raised above 260 °C, the hydrogarnet is broken down and the oxides recombine as  $2\text{CaO} \cdot \text{Al}_2\text{O}_3 \cdot \text{SiO}_2 \cdot \text{H}_2\text{O}$ . A similar sequence of reactions presumably occurs when gehlenite or the mixed oxides are used as starting materials. Complete conversion was never attained, as even the best preparation of the new hydrate contained a small amount (perhaps 5 percent) of hydrogarnet.

The new compound may be called a gehlenite hydrate on the basis of its stoichiometry, despite the indications that the gehlenite does not hydrate directly but goes through the hydrogarnet stage.

Compositions of compounds and mixtures given hydrothermal treatment are shown in relation to the anhydrous system  $\text{CaO} \cdot \text{Al}_2\text{O}_3 \cdot \text{SiO}_2$ . Filled circles indicate that the new phase ( $2\text{CaO} \cdot \text{Al}_2\text{O}_3 \cdot \text{SiO}_2 \cdot \text{H}_2\text{O}$ ) was observed in product. Open circles indicate that the new phase was not observed. Insert: Crystals of gehlenite hydrate average about 10 microns on an edge.





# Standards and Calibration

## Impedance Measurements of Coaxial Components Extended to 4 GHz

The NBS Radio Standards Laboratory (Boulder, Colo.) announces that two-terminal impedance measurements of coaxial components are now available over a range of 50 MHz to 4 GHz. Formerly these calibration services were available only to 1 GHz.

In many applications not involving high power, the use of coaxial systems at frequencies above 1 GHz has become popular from the standpoint of economy, flexibility, and smaller physical size compared with rigid waveguide systems. This, together with improved coaxial connectors, has created a need for accurate measurements on coaxial systems at higher frequencies. In response to this need, NBS has extended calibration services for two-terminal impedance measurements on coaxial components to 4 GHz.

Uncertainties of measurement vary, depending upon the quality of coaxial connectors of the items being calibrated. However, for connector pairs having a VSWR of  $(1.001 + 0.001 \times \text{frequency in GHz})$  with reference to 50 ohms, the following uncertainties of measurement apply where the VSWR of the calibrated item is equal to or less than 2:

VSWR: From  $\pm 0.005$  at 1 GHz, to  $\pm 0.010$  at 4 GHz

Impedance magnitude: From  $\pm 0.5$  percent at 1 GHz, to  $\pm 1$  percent at 4 GHz

Phase angle: From  $\pm 0.5^\circ$  at 1 GHz, to  $\pm 1.0^\circ$  at 4 GHz.

Calibration uncertainties for frequencies below 1 GHz remain unchanged.

## Metrologia

Appearing less than two months after the first, the second issue (dated April 1965) of *Metrologia*, a quarterly devoted to scientific metrology, contains articles of interest to four principal areas of precise measurement: optical metrology, and the measurement of fundamental electrical quantities, high pressures, and high temperatures. It also inaugurates a "Letters to the Editor" department.

The new journal is published under the auspices of the International Committee of Weights and Measures. Its editorial aims and the contents of its first number were described in the June issue of this Bulletin.<sup>1</sup>

Guy Lansraux outlines, in the first article, the technique of "diffrimoscropy," in which an image is formed out of coherent light diffracted at the border of an opaque object. The characteristics of such images are described and illustrated, with indications of how they can be (1) used to study the interaction of matter

and light and (2) applied in dimensional metrology. The instrument described is said to surpass in performance the conventional profile projector. A later paper will deal with the theory of the process.

A fairly detailed account is given by W. K. Clothier of a calculable standard of capacitance constructed at the National Standards Laboratory of Australia. The standard is the change in capacitance produced when one screening electrode is displaced with respect to another. The displacement is measured by a built-in Fabry-Perot interferometer, and the maximum change in capacitance is 0.25 pF. It is estimated that the standard makes possible capacitance measurements accurate to 1 part in  $10^7$  if electrostatic units are employed; but for electromagnetic units this figure must be reduced because of the uncertainty in the value for the speed of light.

Recent developments in the accurate measurement of steady high pressures by means of free piston gages at the National Physical Laboratory, Teddington, England, are described by R. S. Dadson, R. G. P. Grieg, and Angel Horner. Major attention is given to determinations by two different experimental methods of the pressure-dependent elastic changes in effective cross-section area of the piston-cylinder assemblies. It is concluded that the change in area is sensibly proportional to the pressure, and that the constant of proportionality could be measured with an uncertainty of  $\pm 2$  percent, corresponding to an uncertainty in effective area of  $\pm 1$  part in  $10^5$  at 1,000 bars, increasing in proportion to pressure at higher pressures.

The fourth article, on gas thermometry at high temperatures, is by Helmut Moser of the Physikalisch-Technische Bundesanstalt, Braunschweig, Germany; it is in German, with English abstract. A short description is given of the principal types of gas thermometer with their advantages and disadvantages and there follows a discussion of the important sources of systematic error: specifically, those due to indirect determination of fixed-point temperatures, uncertainty of the expansion coefficient of the bulb material or of the second virial coefficient of the gases, and desorption effects. Recent gas thermometer results are compared with those obtained by the optical method.

In the Letters to the Editor, Chester H. Page of the NBS Institute for Basic Standards, discusses the proposal to define the ampere in terms of the gyromagnetic ratio of the proton; and M. Romanowski of the Division of Applied Physics, National Research Council of Canada, reports some instances of repeated measurements of a fixed physical quantity which all differed in much the same systematic way from the Gaussian law.

<sup>1</sup> NBS Tech. News Bull. 49, 93 (June 1965). It is reported, as this goes to press, that the number of subscribers has already risen to 440.

# PUBLICATION and CONFERENCE

## Briefs

### First Volumes of Humidity and Moisture Series

The effects of humidity, not only on comfort but also on materials and processes, was the general topic of papers delivered at the 1963 International Symposium on Humidity and Moisture. Most of these papers are now being published in a four-volume set entitled *HUMIDITY and MOISTURE Measurement and Control in Science and Industry*. Editor in chief of the series is Arnold Wexler of the NBS Institute for Basic Standards.

The wide concern with humidity is indicated by the sponsorship of the Symposium, which was held at Washington, D.C., in May 1963. The sponsors were the National Bureau of Standards; the U.S. Weather Bureau; the American Society of Heating, Refrigeration, and Air-Conditioning Engineers; the American Meteorological Society; and the Instrument Society of America.

The first two volumes of the set are now available and can be obtained from the publisher, Reinhold Publishing Corp., New York. The first volume, *Principles and Methods of Measuring Humidity in Gases*, edited by Robert E. Ruskin of the U.S. Naval Research Laboratory, costs \$30. Methods mentioned in this volume range from the hair hygrometer to advanced and specialized methods devised more recently. Techniques of interest to scientists desiring to measure humidity in gases are described under the heading: Psychrometry, Dew-Point Hygrometry, Electrical Hygrometry, Spectroscopic Hygrometry, Coulometric Hygrometry, and Miscellaneous Methods.

The second volume, entitled *Applications*, was edited by Elias J. Amdur of Honeywell, Inc., and is now available from the publisher at a cost of \$27.50. It deals with the application of humidity in the general fields of biology and medicine, agriculture, environmental chambers, air conditioning, process control, meteorology, and atmospheric refraction. Of particular interest are the uses of controlled humidity in medical treatment, in harvesting cotton, and in obtaining maximum dairy production.

The two remaining volumes of the series, *Fundamentals and Standards* and *Principles and Methods of Measuring Moisture in Liquids and Solids*, have just been published and will be reviewed in the next Bulletin.

### Critical Points Conference

A Conference on Phenomena in the Neighborhood of Critical Points, attended by 191 American and foreign physicists, was held at the Bureau on April 5-8, 1965. The purpose of the Conference was to bring together theorists and experimentalists, who are studying critical phenomena in a variety of systems, in order to discuss the successes and shortcomings

of current theory and experimental techniques, and to exchange ideas on new approaches. The Conference was aided by grants from the National Science Foundation and the Office of Naval Research.

The Conference participants included 29 from Western Europe, 3 from Japan, 3 from Canada, 1 from Australia, and 1 from Israel. Among the noted scientists who took part in the Conference were George E. Uhlenbeck, former president of the American Physical Society; William Fairbank, Research Corporation Prize winner; Peter J. Debye, Nobel Prize winner in chemistry in 1936; and Chen N. Yang, Nobel Prize winner in physics in 1958.

The Conference opened with a welcoming address by Robert A. Huntton, Director of the NBS Institute for Basic Standards. He emphasized the role that the Conference would play in stimulating the work of Bureau scientists studying critical phenomena.

Over 35 papers were given during the first 3 days of the Conference. The following topics were covered: equilibrium critical phenomena in fluids, critical phenomena in ferro and antiferro magnets, logarithmic singularities, elastic and inelastic scattering, and transport and relaxation phenomena.

The last day of the Conference, a program was scheduled to permit further discussion of the developments of the Conference, and to allow consideration at length of new theoretical ideas and approaches.

The proceedings of the Conference will be published in late summer.

### Millimeter Wave and Far Infrared Conference

The Bureau's Radio Standards Laboratory is joining with the Institute of Electrical and Electronics Engineers (G-MTT), the International Scientific Radio Union, and the University of Colorado in sponsoring a Millimeter Wave and Far Infrared Conference. It will be held August 30 to September 1 at the Stanley Hotel, Estes Park, Colo., not far from the Boulder Laboratories.

The Conference will bring together engineers and physicists to review developments and applications in this field. Topics to be discussed will be: Generation, detection, transmission, astronomy, spectrographic techniques, quantum phenomena and masers, measurement techniques, and systems. These areas will be studied or exploited in the foreseeable future at the frequencies covered by the Conference.

The program and registration form for the Conference are available now. Arrangements are being made for publication of papers presented at the Conference. Further inquiries about the Conference should be made to: Bureau of Continuation Education, 328 University Memorial Center, University of Colorado, Boulder, Colo., 80304.



# Publications of the National Bureau of Standards

## Periodicals

- Technical News Bulletin*, Volume 49, No. 6, June 1965. 15 cents. Annual subscription: \$1.50. 75 cents additional for foreign mailing. Available on a 1-, 2-, or 3-year subscription basis.
- IRPL Ionospheric Predictions* for September 1965. Three months in advance. Number 30, issued June 1965, 25 cents. Annual subscription: \$2.50, 75 cents additional for foreign mailing. Available on a 1-, 2-, or 3-year subscription basis.
- Journal of Research of the National Bureau of Standards*
- Section A. *Physics and Chemistry*. Issued six times a year. Annual subscription: Domestic, \$4; foreign, \$4.75. Single copy, 70 cents.
- Section B. *Mathematics and Mathematical Physics*. Issued quarterly. Annual subscription: Domestic, \$2.25; foreign, \$2.75. Single copy, 75 cents.
- Section C. *Engineering and Instrumentation*. Issued quarterly. Annual subscription: Domestic, \$2.25; foreign, \$2.75. Single copy, 75 cents.
- Section D. *Radio Science*. Issued monthly. Annual subscription: Domestic, \$9; foreign, \$11.50. Single copy, \$1.00.

## Current Issues of the Journal of Research

- J. Res. NBS 69A (Phys. and Chem.)*, No. 4 (July-August 1965).

Correlation of successive atomic steps in crystals by relaxation mode analysis. A. D. Franklin.

Reduction of crystallographic point groups to subgroups by homogeneous stress. H. S. Peiser and J. B. Wachtman, Jr.

Effect of hydrostatic pressure on the refractive indices of some solids. R. M. Waxler and C. E. Weir.

Crystallization kinetics and polymorphic transformations in polybutene-1. J. Powers, J. D. Hoffman, J. J. Weeks, and F. A. Quinn, Jr.

Compliance-time-temperature relationships from indentation measurements on a pure-gum rubber vulcanizate. F. L. Roth, G. W. Bullman, and L. A. Wood.

Distribution function of the end-to-end distances of linear polymers with excluded volume effects. J. Mazur.

An additivity rule for the vapor pressure lowering of aqueous solutions. R. A. Robinson and V. E. Bower.

Frank-Condon factors to high vibrational quantum numbers V: O<sub>2</sub> band systems. R. W. Nicholls.

- An absolute temperature scale from 4 °K to 20 °K determined from measurements with an acoustical thermometer. H. H. Plumb and G. Cataland.

- J. Res. NBS 69C (Engr. and Instr.)*, No. 3 (July-Sept. 1965).
- Two-terminal dielectric measurements up to  $6 \times 10^8$  Hz. M. G. Broadhurst and A. J. Bur.

Improved ten-picofarad fused silica dielectric capacitor. R. D. Cutkosky and L. H. Lee.

Errors in the series-parallel buildup of four-terminal resistors. C. H. Page.

Centerable rotator for measuring properties of crystals. C. P. Saylor and H. B. Lowey.

Equipment for single-crystal growth from the melt suitable for substances with a low melting point. A. T. Horton and A. R. Glasgow.

Phase and amplitude contrast microscopy in partially coherent light. M. De and P. K. Mondal.

Exposure time relations for Kossel microdiffraction photographs. H. Yakowitz and D. L. Vieth.

Cartesian diver as a density comparator. H. A. Bowman and R. M. Schoonover.

Cryogenic behavior of selected magnetic materials. J. J. Gnielwek and E. Ploge.

- Radio Sci. J. Res. NBS/URSI 69D*, No. 7 (July 1965).

International comparison of atomic frequency standards via VLF radio signals. A. H. Morgan, E. L. Crow, B. E. Blair.

Control of WWV and WWVH standard frequency broadcasts by VLF and LF signals. B. E. Blair and A. H. Morgan.

Measurements of the total electron content and the equivalent slab thickness of the midlatitude ionosphere. R. V. Bhonsle, A. V. da Rosa, and O. K. Garriott.

D-region absorption at 10 and 15 Mc/s during the total solar eclipse of July 20, 1963. G. M. Lerfeld, J. K. Hargreaves, and J. M. Watts.

Effect of the eclipse of 20 July 1963 on VLF signals propagating over short paths. J. H. Crary and D. E. Schneible.

A comparison of radar auroral reflection data with acoustic wave theory. R. L. Leadabrand.

Electromagnetic properties of a plasma covered antenna. D. J. Jacavano.

Influence of an inhomogeneous ground on the propagation of VLF radio waves in the earth-ionosphere waveguide. J. R. Wait.

Aspects of the terrestrial ELF noise spectrum when near the source or its antipode. L. G. Abraham, Jr.

Analysis of linear arrays focused in the Fresnel region. P. P. Lombardini, R. Doviak, and J. Goldhirsh.

Theory of coil antennas. T. Padhi.

Small prolate spheroidal antenna in a dissipative medium. R. H. Williams, R. D. Kelly, and W. T. Cowan.

Calculated curves for groundwave propagation over inhomogeneous earth with pronounced topographical features. K. Furutsu.

## Other NBS Publications

Ionospheric radio propagation, K. L. Davies, NBS Mono. 80 (Apr. 1965), \$2.75.

Examination of liquefied petroleum gas liquid-measuring devices, M. W. Jensen, NBS Handb. 99 (Apr. 23, 1965), 35 cents.

Systems engineering in ceramics. Proceedings of a symposium April 19, 1964, NBS Misc. Publ. 267 (May 1, 1965), \$2.00.

Steel medicine cabinets, NBS CS267-65 (Mar. 1, 1965), 10 cents.

Hide trim pattern for domestic cattlehides, NBS CS268-65 (Apr. 4, 1965), 10 cents.

Research on crystal growth and characterization at the National Bureau of Standards July to December 1964, Ed. H. F. Murdie, NBS Tech. Note 260 (May 8, 1965), 50 cents.

Studies of solar flare effects and other ionospheric disturbances with a high frequency Doppler technique, V. Agy, D. M. Baker, and R. M. Jones, NBS Tech. Note 306 (Apr. 28, 1965), 75 cents.

Phase and amplitude diversity in over-water transmissions at two microwave frequencies, H. B. Janes, A. W. Kirkpatrick, D. M. Waters, and D. Smith, NBS Tech. Note 307 (Apr. 12, 1965), 40 cents.

Data reduction for stable auroral red arcs observed at Rapid City, South Dakota, J. E. Cruz, R. Davies, L. K. Dippleman, E. Marovich, L. R. Megill, M. H. Rees, L. Reisbeck, and F. E. Roach, NBS Tech. Note 308 (May 3, 1965), 50 cents.

A bibliography of experimental saturation properties of the cryogenic fluids, N. A. Olien and L. A. Hall, NBS Tech. Note 309 (Apr. 9, 1965), 60 cents.

Attenuation of the ground wave of a low frequency electromagnetic pulse, J. C. Morgenstern and J. R. Johler, NBS Tech. Note 310 (Apr. 1965), 30 cents.

## Publications in Other Journals

This column lists all publications by the NBS staff, as soon after issuance as practical. For completeness, earlier references not previously reported may be included from time to time.

### Chemistry:

Gas-phase photolysis of methyl radicals with added organic compounds, R. D. Doepker and P. Ausloos, *J. Chem. Phys.* 41, No. 6, 1865-1871 (Sept. 15, 1964).

Effect of particle shape and size distribution in a reinforced polymer, R. L. Bowen, *J. Am. Dental Assoc.* 69, 481-495 (Oct. 1964).

Growth of oxalic acid single crystals from solution: solvent effect on crystal habit, J. L. Torgensen and J. Strassburger, *Science* 146, No. 3640, 53-55 (Oct. 2, 1964).



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Infrared studies of the photolysis of  $\text{HN}_3$  in inert and reactive matrices. The infrared spectrum of  $\text{NH}_3$ , D. E. Milligan and M. E. Jacox, *J. Chem. Phys.* **49**, No. 9, 2838-2841 (Nov. 1, 1964).

The solubility of water vapor in molten alumina, J. J. Diamond and A. L. Dragoo (Proc. Conf. Arthur D. Little, Inc., Cambridge, Mass., Oct. 4-5, 1962), Book, Thermal Imaging Techniques, ed. P. E. Glaser and R. F. Walker, Ch. 17, pp. 225-228 (Plenum Press Inc., New York, N.Y., 1964).

**Engineering and Instrumentation:**

A study of the variation of the surface electrical resistance of lead iodide films with relative humidity at room temperature, F. E. Jones (Proc. 1963 Intern. Symp. Humidity and Moisture, Washington, D.C.), Book, Humidity and Moisture. Measurement and Control in Science and Industry. Vol. 1, Principles and Methods of Measuring Humidity in Gases, Sec. III, Paper 37, pp. 361-365 (Reinhold Publ. Corp., New York, N.Y., 1965).

An investigation of psychrometric measurement techniques in air-conditioning calorimetry, J. C. Davis and P. R. Achenbach (Proc. 1963 Intern. Symp. Humidity and Moisture, Washington, D.C.), Book, Humidity and Moisture. Measurement and Control in Science and Industry. Vol. 2, Applications, Sec. IV, Paper 37, p. 303-313 (Reinhold Publ. Corp., New York, N.Y., 1965).

Application of cooled air detector in an optical system, S. Abramowitz, A. M. Bass, and A. E. Ledford, Jr., *Appl. Opt.* **4**, No. 2, p. 255 (Feb. 1965).

Calculation of the temperature of a flat-plate wet surface under adiabatic conditions with respect to the Lewis relation, T. Kusuda (Proc. 1963 Intern. Symp. Humidity and Moisture, Washington, D.C.), Book, Humidity and Moisture. Measurement and Control in Science and Industry. Vol. 1, Principles and Methods of Measuring Humidity in Gases, Sec. I, Paper 2, p. 16-32 (Reinhold Publ. Corp., New York, N.Y., 1965).

**Mathematics:**

A uniqueness theorem for entire functions, R. F. DeMar, *Proc. Am. Math. Soc.* **16**, No. 1, 69-71 (Feb. 1965).

Nomograms for computing real, imaginary, and absolute values of vector ratios, H. S. Bowman, *Acoust. Soc. Am.* **37**, No. 4, 751 (Apr. 1965).

On a theorem concerning existence of interpolating functions, R. F. DeMar, *Trans. Am. Math. Soc.* **114**, No. 1, 23-29 (Jan. 1965).

**Metrology:**

A study of errors in the measurement of microscopic spheres, C. P. Saylor, *Appl. Opt.* **4**, No. 4, 477-486 (Apr. 1965).

Definition of "ampere" and "magnetic constant," C. H. Page, *Proc. IEEE* **53**, No. 1, 100-101 (Jan. 1965).

Electromagnetic measurement, H. W. Lance, *SPACE/AERONAUTICS Res. Develop. Handb.* **42**, No. 4, 199-202 (1964-65).

**Physics:**

18-cm spectrum of OH, H. E. Radford, *Phys. Rev. Letter* **13**, No. 17, 534-535 (Oct. 26, 1964).

Low temperature magnetic transitions in lanthanum trichlorides, *Phys. Letters* **12**, No. 1, p. 5 (Sept. 1, 1964).

An investigation of plasma boundaries with electromagnetic surface waves, H. W. Wassink and A. J. Estlin, *J. Appl. Phys.* **35**, 2795-2800 (Oct. 1964).

Modulus of rupture of glass in relation to fracture pattern, M. J. Kerper and T. G. Scuderi, *Am. Ceram. Soc. Bull.* **43**, No. 9, 622-625 (Sept. 7, 1964).

**Radio Science:**

Cavity resonances for spherical earth with a concentric anisotropic shell, J. R. Wait, *J. Atmos. Terres. Phys.* **27**, 81-89 (1965).

On the effect of solar disturbances on the low-radiofrequency ionosphere reflection process, J. R. Jöhler and J. D. Harper, Jr. (North Atlantic Treaty Organization Advisory Group for Aeronautical Research and Development. Avionics Panel. 6th Symp. Ionospheric Research Committee, AGARD, Naples, Italy, 1961), Book, The Effect of Disturbances of Solar Origin on Communications, ed. G. J. Cassman, p. 303-304 (Pergamon Press, Inc., New York, N.Y., 1963).

Potential use of passive probing of atmospheric structure by thermal emissions at radio frequencies, B. R. Bean, E. R. Westwater and R. L. Abbott (Proc. 1963 Intern. Symp. Humidity and Moisture, Washington, D.C.), Book, Humidity and Moisture. Measurement and Control in Science and Industry. Vol. 2, Applications. Sec. VII, Paper 71, pp. 595-608 (Reinhold Publ. Corp., New York, N.Y., 1965).

Radio refractometry and its potential for humidity studies, R. E. McGavin and M. J. Vetter (Proc. 1963 Intern. Symp. Humidity and Moisture, Washington, D.C.), Book, Humidity and Moisture. Measurement and Control in Science and Industry. Vol. 2, Applications, Sec. VII, Paper 66, pp. 553-560 (Reinhold Publ. Corp., New York, N.Y., 1965).

The use of the radio refractometer to measure water vapor turbulence, B. R. Bean and R. E. McGavin (Proc. 1963 Intern. Symp. Humidity and Moisture, Washington, D.C.), Book, Humidity and Moisture. Measurement and Control in Science and Industry. Vol. 2, Applications, Sec. VII, Paper 67, pp. 561-568 (Reinhold Publ. Corp., New York, N.Y., 1965).

*Publications for which a price is indicated are available by purchase from the Superintendent of Documents, U.S. Government Printing Office, Washington, D.C., 20402 (foreign postage, one-fourth additional). Reprints from outside journals and the NBS Journal of Research may often be obtained directly from the authors.*

**Patents**

*The following U.S. Patents have recently been granted on NBS inventions and, except as noted, are assigned to the United States of America as represented by the Secretary of Commerce:*

3,164,782 January 5, 1965. Gas-filled envelope for solid laser tube having internal electrodes. Fred D. Ordway, Jr. (Licensed to the United States of America as represented by the Secretary of Commerce.)

3,166,725 January 19, 1965. Broadband tuning transformer permitting independent matching at adjacent frequencies. Glenn F. Engen.

3,167,244 January 26, 1965. Automatic or manual multiple programmer. Anthony A. Berlinsky and Martin J. Brennan.

3,167,770 January 26, 1965. Time-multiplex system for the distribution of serial pulse time codes with microsecond synchronization. Robert H. Doherty and Thomas L. Davis. AIR FORCE.)

3,174,710 March 23, 1965. Take-off safety indicator. Harold D. Hoekstra. (Licensed to the United States of America as represented by the Secretary of Commerce.)

3,177,017 April 6, 1965. Ball and socket pipe joint. Leslie A. Guildner and Harold F. Stimson.

3,177,421 April 6, 1965. Rectified alternating-current converter. George Franklin Montgomery.

3,179,623 April 20, 1965. Method of preparing a monomer having phenoxy and methacrylate groups linked by hydroxy glyceryl groups. Rafael L. Bowen.

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